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Calcium fortification of roasted and ground coffee with different calcium salts

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ABSTRACT. This study determined the calcium content and sensory attributes of roast and ground coffee fortified with calcium carbonate, calcium citrate malate, and calcium phosphate. The beverages made from fortified coffee powder were compared with a control (unfortified beverage). Two experts in coffee sensory testing analyzed six samples of coffee with the addition of different calcium salts; they assessed the aroma, color, and characteristic flavor of traditional coffee. The formulations containing calcium carbonate (A), calcium citrate malate (B), and calcium phosphate (C) were selected and used to verify the effect of using paper and polyester to filter infusions. Formulation B had the greatest purchase intent and presented the highest calcium content in the beverage, and it was considered rich in this mineral. The daily habit of drinking a cup of coffee (50 mL) has been popularized in Brazil; therefore, the addition of calcium citrate malate into traditional coffee powder can be an excellent calcium source and ensure the healthy intake of this macro mineral.

Keywords: beverage, calcium carbonate, calcium citrate malate, calcium phosphate, filters, sensory analysis.

Fortificação de cálcio em café torrado e moído com diferentes sais de cálcio

RESUMO. O estudo determinou o conteúdo de cálcio e os atributos sensoriais no pós de café torrado e moído fortificado com carbonato de cálcio, citrato malato de cálcio e fosfato de cálcio. As bebidas de café torrado e moído fortificadas foram comparadas com uma bebida não fortificada controle. Dois especialistas em prova de café analisaram sensorialmente seis cafés adicionados de distintos sais de cálcio quanto ao odor, cor e sabor característico do café tradicional. Posteriormente as formulações contendo carbonato de cálcio (A), citrato malato de cálcio (B) e fosfato de cálcio (C) foram selecionados e verificados o efeito do uso de coador de papel e poliéster nas infusões. A formulação B apresentou maior teor de cálcio na bebida, sendo classificada como rica neste metal e obteve maior intenção de compra. Tendo em vista a popularização do hábito de se ingerir uma xícara de café (50 mL) diariamente no Brasil, a adição de citrato malato de cálcio no pó de café tradicional, pode ser uma excelente fonte cálcio e garantir a ingestão saudável deste macromineral.

Palavras-chave: bebida, carbonato de cálcio, citrato malato de cálcio, fosfato de cálcio, coadores, análise sensorial.

Introduction

Calcium is one of the most important dietary factors for bone metabolism. A great deal of scientific literature has shown that high dietary calcium intake and its bioavailability are associated with the reduced risk of osteoporosis, hypertension, colon cancer, kidney stones, stroke, obesity, lead absorption, and premenstrual syndrome (MCCARRON; HEANEY, 2004; UNRUH VON et al., 2004). Unfortunately, there are few excellent dietary sources of calcium, especially if milk and dairy products are not a regular part of the diet (CERKLEWSKI, 2005). Fortification of foods such as orange juice, carbonated beverages, yeast breads, and breakfast cereals has been used to help improve calcium intake (BABARYKIN et al., 2004; FAIRWEATHER-TAIT; TEUCHER 2002).

For its specific flavor and positive effects on humans, coffee is one of the most popular beverages in the world. The average citizen of the European Union consumes 4.96 kg of coffee per year (ICO, 2008). Coffee is the most consumed beverage in Brazil, second only to water. Coffee consumption in Brazil increased by 3% from 2011 to 2012, but this increase was provided by the Brazilian Coffee Industry Association (ABIC). In addition, the *per capita* consumption was 6.18 kg of grain per year; this is equivalent to 4.94 kg of roasted coffee, approximately 83 liters for each Brazilian. This value

represents an increase of 1.23% over the previous period of 2010. Thus, it is possible to affirm that Brazilians consume various forms of coffee such as latte, cappuccino, espresso, and other combinations (SOLOMON, 2002).

Several commercial calcium salts have been used for calcium enrichment of milk/beverages, e.g., calcium carbonate, calcium chloride, calcium phosphate, tribasic calcium phosphate, calcium citrate malate, calcium lactate, calcium gluconate, calcium lactate gluconate, and natural milk calcium (GOLDSCHER; EDELSTEIN, 1996; TATEO et al., 1997).

Calcium-fortified foods should have similar physical and sensory characteristics as their non-fortified counter parts (FAIRWEATHER-TAIT; TEUCHER, 2002). This study determined calcium contents and sensory attributes of all-purpose roasted and ground coffee fortified with calcium carbonate, calcium citrate malate, and calcium phosphate. Beverages of roasted and ground coffee fortified were compared to non-fortified control beverages.

Material and methods

Addition of calcium salts in roast and ground coffee

The experiments were performed in the Itamaraty Roasting Coffee Industry, Rolândia, Paraná State, Brazil. The roast and ground coffee Itamaraty® was used in this study. Calcium addition tests were performed on roasted and ground coffee using the calcium salts that had good water solubility. Six calcium salts were used: tricalcium phosphate ($Ca_3O_8P_2$); monocalcium phosphate ($CaH_4P_2O_8$); calcium carbonate ($CaCO_3$); calcium lactate ($Ca\ (CH_3CHOHCOO)_2\ 5H_2O$); calcium citrate malate ($C_4H_40_5Ca$), and Calci-K® (calcium compound - $C_6H_8O_7$, $Ca\ (OH)_2$, H_3PO_4 and KOH), at the following levels: 0.75, 1.88, 0.78, 2.24, 1.50 and 1.66 g 100 g^{-1} of coffee powder, respectively. Calcium salts were added in 500 g of roast and ground coffee with further homogenization in a closed, white, and odorless container, in the test laboratory of the industry.

Selection of roast and ground coffee containing calcium salts according to similarity to traditional coffee

The opinions from two experts in coffee tasting were requested in the selection of the coffee with calcium salt with greater similarity to traditional coffee directly in the Itamaraty® Coffee Company. Initially, six calcium salts, tricalcium phosphate, monocalcium phosphate, calcium carbonate, calcium lactate, calcium citrate malate, and Calci-K®, were added to coffee, and the beverages were prepared with paper filters. The samples were presented in pairs, one with calcium salt and the other with traditional coffee. The expert panelists then had to opine on the similarity of the beverages according to the attributes: aroma, color, and characteristic flavor of traditional coffee.

The preparation of beverages followed the manufacturer's recommendations (Itamaraty®), consisting of 80 g of powder to 1 L of water. The water temperature was maintained at 90 to 100°C and poured slowly to moisten the powder, and the contact time ranged from 4 to 6 min. The tests using polyester filters were performed by washing them with water and reusing them after each preparation. The paper filters were discarded after use.

Subsequently, the experts selected coffee beverages with three different calcium salts. The selected samples were evaluated by potential consumers to assess their acceptability, and the calcium contents were quantified in coffee preparations.

Physical and physico-chemical analysis

The colors of coffee powders added with calcium salts were measured using Agtron disks. The pH of the beverages was measured at 25°C using a pH meter (Quimis, Q400AS). The particle size analysis was performed using a sieve of 30-mesh, and the retained powder percentage was determined. The moisture content was determined according to Cunniff (1998).

Sensory analysis

Besides the opinions of experts in choosing the best coffee, sensory analysis of acceptance and purchase intent were performed. These analyses allowed to collect data about traditional coffee consumption and confirm the consumers approval for coffee. In this context, the acceptance test was conducted with the three beverages, A, B, and C, containing the following calcium salts: calcium carbonate, calcium citrate malate and tricalcium phosphate, respectively. According to the experts, they were closer to traditional coffee.

The preparation of coffee with calcium salts (powder and beverage) was followed as described above, with 2% added sugar in order to simulate the way the beverage is commonly consumed.

In the affective test of acceptance, the overall aspect was considered for the formulations. The group of panelists was comprised 50 volunteers; untrained and possible potential consumers of traditional coffee, they evaluated the samples using a structured hedonic scale of 10 points (1 = extreme

dislike to 10 = extreme like; ABNT, 1993; ABNT 1998). The samples were presented in a randomized complete block design in order to highlight the comparison between formulations. A 5-point scale was used for the purchase intention test in order to verify whether people would buy the formulations developed (1 = certainly would not buy; 5 = certainly would buy) (LAWLESS; HEYMANN, 2010).

Hygienic and sanitary evaluation during manipulation of beverages

During the preparation of coffee formulations - mixture of salts and beverages - for sensory analysis, good practices of manufacturing were adopted. In order to ensure product safety and minimize the risk of cross-contamination, all utensils and surfaces were evaluated for the amount of adenosine triphosphate (ATP). This technique aims to evaluate the energy present in the cells. The measurements were performed in a Luminometer apparatus (Uni-lite NG) by the amount of light emitted in the reaction of luciferin/sample/luciferase, estimating the amount of biological material present in the sample. The standard adopted by the company (Itamaraty®) is 200 relative light units (RLU).

Ethical aspects

This study was approved by the Standing Committee on Ethics in Research Involving Humans of the Institution, untrained volunteers (students and employers), for the performance of sensory tests, under the protocol CAAE no. 14866813.3.0000.5547.

Quantification of the calcium content

The quantification of calcium in roast and ground coffee (powder and beverage) was initiated with the digestion of the sample by the wet method using nitric, perchloric, and hydrochloric acids. The mixture was kept at room temperature (25°C) for approximately 12h and then digested at 100°C for 4 hours (AOAC, 1995). Sample analyses were conducted in an Atomic Absorption Spectrometer Model Nova 300 (Analytik Jena AG, Germany), with a wavelength of 239.9 nm and spectral bandwidth of 0.2 nm. Flame gases used were acetylene and nitrous oxide. For quantification of calcium, the method of external standard with calibration curves was applied, and the results were expressed in mg 100 g^{-1} for the powder form and in mg L^{-1} for the beverage.

Calculation of daily reference intake

Calcium salts were added in 500 g of traditional roast and ground coffee at two levels, corresponding to 15 and 30% of the Daily Reference Intake (DRI) recommended for calcium. These levels correspond to 150 and 300 mg of Ca per 100 g^{-1}, respectively, of coffee powder (BRASIL, 1998).

Statistical analysis

The acceptance test results were submitted to analysis of variance (ANOVA) with two-factor without replication (sample and panelist) and the means were compared using the "post-hoc" Tukey test. The software used in this analysis was Statistica, version 8.0 (STATSOFT, 2007). The significance level for rejection of the null hypothesis adopted in this analysis was 5% ($p < 0.05$).

Results and discussion

During the roasting stage, at the roasting degree verification, the experts did not notice changes in the coffee. The samples were in the range of "medium light" to "almost medium light" units (Agtron75) and "moderately dark" to "medium light" (Agtron 45). The first classification is characterized by "light brown" grain, "light body", minimal aroma, flavor similar to tea, and no surface oil. The second classification is characterized by a grain color tending to a "more intense brown", "heavy body", lower acidity, surface oil, and a noticeable sweet bitterness (MELO, 2004). After grinding the roast coffee, changes in the hue of the product with calcium carbonate added were not observed. The hypothesis for the color change is based on the particle size (very small) and color (very light). The average moisture content of the roast and ground coffee was 3.02% on a wet basis. According to Brasil (2005a), the moisture content allowed for this type of product is 5% (g 100 g^{-1}). In the particle size analysis, all formulations showed less than 30% of retention on the 30-mesh sieve; therefore, they were classified as traditional coffee. This analysis defined an infusion time of 4 to 6 minutes for both types of filters - paper and polyester.

After the addition of calcium salts, the pH value of beverages ranged from 5.0 to 6.0. Values of 5.1 to 5.2 were determined in a study by Scholz et al. (2013) in coffee cultivars from the Agronomic Institute of Paraná - Iapar. Mamede et al. (2010) obtained for soluble decaffeinated coffee pH = 4.9-5.2. In this context, the incorporation of different calcium salts leads to very little change in the pH of the formulations evaluated.

When evaluating the types of coffee filters - polyester and paper - experts found no difference in the traditional coffee prepared with each one of

them. Due to the convenience of their use, the paper filter can be used without the loss of sensory characteristics in this type of infusion. Another factor to be noted was the preparation time of the beverages with calcium salts, it was the same as proposed for traditional coffee (4-6 min.). This finding indicated that powder was not excessively retained on the filter.

According to the opinion of coffee experts, the samples with 15% calcium DRI (BRASIL, 2005a) did not present a difference when compared to the control beverage. On the other hand, the samples added with 30% of calcium DRI caused differences in the opinions of the coffee experts.

The three beverages that presented sensory characteristics very similar to traditional coffee were the ones calcium carbonate (A), calcium citrate malate (B), and tricalcium phosphate (C) added. These samples were tasted by untrained panelists and did not present differences ($p = 0.13$) when compared to traditional coffee. This fact showed that supplementation of calcium salts did not adversely affect the overall aspect of the products studied, and the acceptance was "like slightly" (average score: 6). The opinion of the panelists were divergent ($p < 0.001$), and the standard deviation was high (± 1.00). However, this kind of result is very common when sensory tests are conducted directly with the target market (PAGAMUNICI et al., 2014a and b; SOUZA et al. 2014).

The purchase intention test (Figure 1) showed that 26, 30, and 32% of the potential coffee consumers "surely would buy" the products A, B and C, respectively. Formulation C may have a promising potential, because 81% of the panelists affirmed a preference for roast and ground coffee instead of soluble coffee. Besides, 78% of the potential consumers have the daily habit of drinking coffee twice a day.

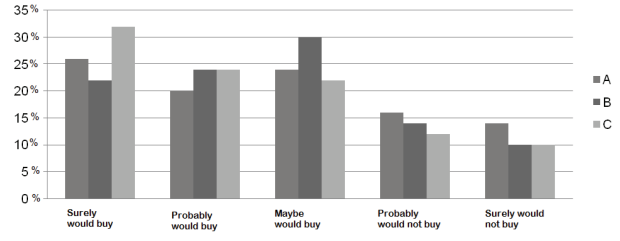

Figure 1. Purchase intention of beverages. A: coffee with calcium carbonate; B: coffee with calcium malate citrate; C: coffee with tricalcium phosphate.

Samples A and B were considered calcium sources and the sample C may be called "rich" in this mineral (Table 1, BRASIL, 1998). The traditional roast and ground coffee presented calcium content similar to that reported in the Brazilian Table of Food Composition Content (TACO, 2011), which is 106.90 mg of calcium per 100 g^{-1} of roast and ground coffee. This content (Table 1) corresponds to 11% of the daily values based on a diet of 2,000 kcal or 8.400 kJ. The difference between results found in Taco (2011) and the sample company (Itamaraty®) may be due to the type of coffee and processing.

Table 1. Amount of calcium in powder of roast and ground coffee.

Calcium salt in powder of traditional coffee[1]	A	B	C	Traditional coffee
Added amount[2]	780.00	1500.00	750.00	-
Média[3]	277.62	211.51	305.13	103.23

[1]mg 100 g^{-1}. [2]mg. [3]Analysis performed in triplicate. A: coffee with calcium carbonate; B: coffee with calcium citrate malate; C: coffee with tricalcium phosphate.

Calcium salts added to the coffee powder had good solubility during the infusion process; this was verified through the contents quantified directly in the beverages (Table 2). The coffee beverage added with calcium citrate malate presented higher contents of calcium; this may be considered indicative of greater solubility of this salt when compared to other calcium salts.

Table 2. Amounts of calcium[1] in the beverages.

Parameters	A	B	C	Traditional coffee
Paper filter	36.96	78.60	36.42	22.84
Polyester filter	23.12	106.68	13.94	17.69

[1]Analysis in triplicate and values expressed in mg L^{-1} of beverage. A: coffee with calcium carbonate; B: coffee with calcium citrate malate; C: coffee with tricalcium phosphate.

The influence of using paper and polyester filters was determinant for the calcium content increasing in the infusions (Table 2). The beverages obtained by the use of paper filter showed an increase of 61.8-77.5% of calcium content. For the polyester filter the increase was 37.9-82.4%. Sample B containing calcium citrate malate had the highest contribution to the increase of calcium content, especially when the polyester filter was used, which presented the best results.

For the calculation of calcium intake, a coffee cup (equivalent to 50 mL) was used as reference. Samples A, B, and C presented 1.84, 3.93, and 1.82 mg, respectively, of calcium per portion. The control sample presented 1.23 mg of calcium in a cup of coffee.

Conclusion

The addition of calcium salts in the powder of ground and roast coffee proved to be promising. Six calcium salts were used in this study; the coffee experts found the beverages with calcium carbonate,

calcium citrate malate, and calcium phosphate as having sensory characteristics similar to traditional coffee. There was no difference in sensory analysis of coffee beverages prepared using paper and polyester filter. However, sample B with calcium citrate malate presented the highest calcium contents and the greatest purchase intention. Calcium citrate malate showed high solubility during infusion, and the beverage with this salt was classified as rich in calcium. In Brazil, the habit of drinking a cup of coffee (50 mL) every day is becoming very popular, it can be in the morning, after meals or during the day activities. Considering this context, the addition of calcium citrate malate in the proportion presented in this study may be an excellent calcium source to ensure the healthy intake of this macromineral.

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